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Lubrication

A Technical Publication Devoted to the Selection and Use of Lubricants

This Issue

ADDITIVES WITH A PURPOSE

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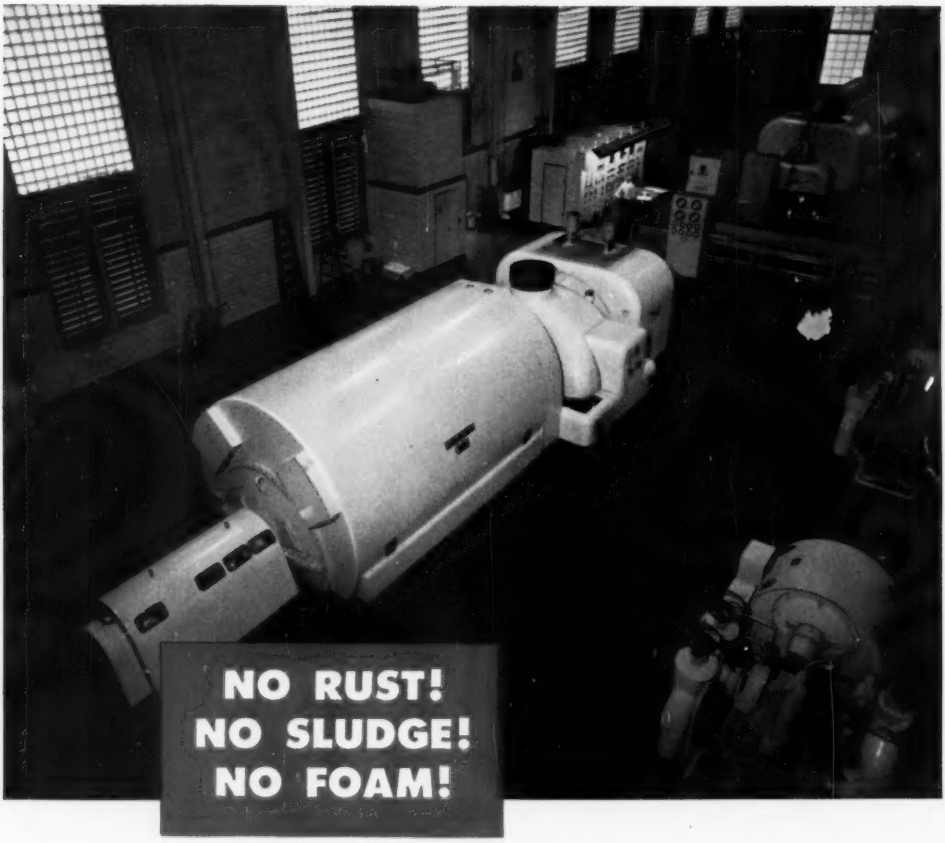

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LUBRICATION

A TECHNICAL PUBLICATION DEVOTED TO THE SELECTION AND USE OF LUBRICANTS

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ADDITIVES WITH A PURPOSE

"WHAT are additives?" For purpose of discussion, the term additives may be defined as chemical compounds which will either enhance or improve some of the properties which a petroleum product already has, or impart new characteristics to it.

For ease of discussion, lubricant additives may be divided into two general classes:

A. Those that affect some physical characteristic of the lubricant such as viscosity index, pour point, foaming, etc.

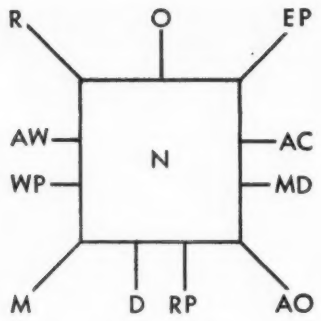
B. Those whose end effect is more chemical in nature and usually more measurable in terms of some performance characteristic. This includes materials such as the oxidation-corrosion inhibitors, detergents and dispersants, extreme pressure and anti-wear agents, oiliness agents, etc.

Much has been written on the general subject of additives during the past ten to fifteen years. In view of the continuous developments in this field, this issue is designed to review the subject further and bring it up to date. Special emphasis is placed on the fact that additives must serve a specific purpose in order to be justified.

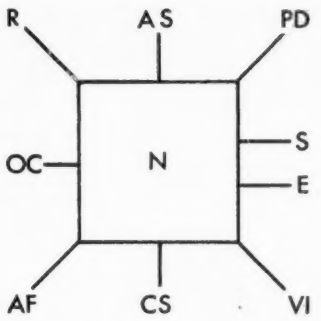
Each of these two classes of additives might be represented by a universal additive molecule as shown in Figure I. In these universal additive molecules, the relative proportion of each component can be varied at will to meet the particular requirement. Thus, by the proper use of these two universal additive molecules, any desired characteristic can be imparted to a lubricant within the limits of additives as they are known today.

These are obviously theoretical additive molecules approaching the ideal situation. If the ideal additive molecules could actually be made, the use of additives in lubricants would indeed be simple. Quite the contrary, the compounding of lubricants with additives to obtain products with the proper characteristics is very complex. Part of this complexity is due to the fact that different chemical compounds are required to impart the particular characteristics desired and since these compounds must be chemically active to accomplish their function, they may affect each other when present in the same medium. These various compounds can be interdependent on each other in different ways such as to complement or supplement each other. It is also possible for any one to affect any or all of the others adversely. The greater the number of types of additives to be used in a lubricant, the more complex becomes the problem of developing it.

There are many hundreds of chemical compounds that have been found to impart or enhance some individual property of a lubricant. However, the fact that these compounds must be compatible with



Chemical Characteristics



Physical Characteristics

Figure 1 — Universal Additive Molecules

N—Nucleus
R—Solubilizing Agent
M—Metal
D—Detergent-Dispersant
AO—Anti-Oxidant
AC—Anti-Corrosive
EP—Extreme Pressure
O—Oiliness
AW—Anti-Wear
MD—Metal Deactivator
RP—Rust Preventive
WP—Water Repellency

N—Nucleus
R—Solubilizing Agent
PD—Pour Depressant
VI—Viscosity Index Improver
AF—Anti-Foam
S—Stringiness
E—Emulsifier
CS—Color Stabilizer
OC—Odor Control Agent
AS—Antiseptic

other chemical addition agents as well as the base lubricant greatly reduces the number of usable ones.

Since additives are chemical compounds, their manufacture is subject to the usual problems common to the chemical industry. This includes problems associated with processing such as materials handling, storage, chemical reactions, mixing, separation, filtration, clarification, distillation, etc.

COMMONLY USED LUBRICANT ADDITIVES

Table I briefly summarizes pertinent information on the commonly used lubricant additives. The following sections will elaborate somewhat on this information, especially on the mechanism involved in order for an additive to accomplish its purpose and some reasons for the use of specific types of additives. Each of the general types of additives will be considered in the order given in this table. This order is not an indication of their relative importance.

OXIDATION-CORROSION INHIBITORS

Since both oxidation and corrosion inhibitors are closely associated to oil oxidation and some chemical addition agents are effective for both purposes, they will be discussed together.

Oil Oxidation

The exact composition of products formed when oil undergoes oxidation is not definitely known because of the complexity and diversity of the hydrocarbons and molecules involved. However, it is generally theorized that organic peroxides are probably the first type of oxidation product formed, since these are invariably found when an oil is oxidized even slightly. These peroxides may act as catalysts to accelerate further oxidation of the hydrocarbons and they are also known to be corrosive to certain bearing metals.

Oxidation based on the peroxide theory may be considered as taking place in three steps: initiation, propagation and termination.

1. Initiation consists of formation of a so-called free radical by the removal of a hydrogen atom from the hydrocarbon molecule. This may be shown schematically as follows, where R represents a hydrocarbon group:

$$\underset{\text{(hydrocarbon molecule)}}{RH} \longrightarrow \underset{\text{(free radical)}}{R' + H'}$$

2. This is followed by a propagation step where the radical (R') can react with oxygen to form the peroxide radical, which in turn can form a hydroperoxide by removing a hydrogen atom from another hydrocarbon molecule.

TABLE I

Commonly Used Lubricant Additives

Type Additive	Type of Compounds Used	Reasons for Use	Mechanism of Action
Anti-oxidants or Oxidation Inhibitors	Organic compounds containing sulfur, phosphorus or nitrogen such as organic amines, sulfides, hydroxy sulfides, phenols. Metals like tin, zinc or barium often incorporated.	To prevent varnish and sludge formation on metal parts. To prevent corrosion of alloy bearings.	Decreases amount of oxygen taken up by the oil thereby reducing formation of acidic bodies. Terminates oil oxidation reactions by formation of inactive soluble compounds or by taking up oxygen. Additive may be oxidized in preference to oil.
Anti-corrosives Corrosion Preventives or Catalyst "Poisons"	Organic compounds containing active sulfur, phosphorus or nitrogen such as organic sulfides, phosphites, metal salts of thiophosphoric acid, and sulfurized waxes.	To prevent failure of alloy bearings by corrosive action. To prevent corrosive attack on other metal surfaces.	Inhibits oxidation so that no acidic bodies are formed or enables a protective film to form on bearings or other metal surfaces. Chemical film formation on metal surfaces decreases catalytic oxidation of the oil.
Detergents	Metallo-organic compounds such as phosphates, phenolates, sulfonates, alcoholates. High molecular weight soaps containing metals like magnesium, barium, calcium, tin.	To keep metal surfaces clean and prevent deposit formation of all types.	By chemical reaction or oxidation direction, oil soluble oxidation products are prevented from becoming insoluble and depositing on various engine parts.
Dispersants	Metallo-organic compounds such as naphthenates and sulfonates. Organic salts containing metals, like calcium, cobalt and strontium.	To keep potential sludge forming insolubles in suspension to prevent their depositing on metal parts.	Agglomeration and deposition of fuel soot and insoluble oil decomposition products is prevented by breakdown into finely divided state. In colloidal form contaminating particles remain suspended in oil.
Oiliness, Film Strength, Extreme Pressure (E.P.) and Anti-Wear Agents	Organic compounds containing chlorine, phosphorus and sulfur such as chlorinated waxes, organic phosphates and phosphites such as tricresyl phosphate and zinc dithiophosphate, and lead soaps such as lead naphthenate.	To reduce friction, prevent galling, scoring and seizure. To reduce wear.	By chemical reaction film is formed on metal contacting surfaces which has lower shear strength than base metal thereby reducing friction and preventing welding and seizure of contacting surfaces when oil film is ruptured.
Rust Preventives	Sulfonates, amines, fatty oils and certain fatty acids, oxidized wax acids, phosphates, halogenated derivatives of certain fatty acids.	To prevent rust of metal parts during shutdown periods, storage or shipment of new or overhauled equipment.	Preferential adsorption of polar type surface active materials on metal surface. This film repels attack of water. Neutralizing corrosive acids.
Metal Deactivators	Complex organic nitrogen and sulfur containing compounds such as certain complex amines and sulfides. Some soaps.	Passify, prevent or counteract catalytic effect of metals on oxidation.	Form inactive protective film by physical or chemical adsorption or absorption. Form catalytically inactive complex with soluble or insoluble metal ions.
Stringiness and Tackiness Agents	Certain high molecular weight polymers and aluminum soaps of unsaturated fatty acids.	Increases adhesiveness of lubricant on metal surfaces, form protective coating.	Increases viscosity of lubricant and imparts adhesive and tackiness characteristics.
Water Repellents	Organosilicon and other polymers, certain higher aliphatic amines and hydroxy fatty acids.	Provide water repellent or resistant properties to non-soap thickened greases and other lubricants.	Surface active agents form protective film on grease thickeners or other components of lubricants to reduce their affinity for water.
Emulsifiers	Certain soaps of fats and fatty acids, sulfonic acids or napthenic acids.	Used to emulsify soluble oils with water to give coolant lubricant type fluid.	Surface active chemical agents reduce interfacial tensions so oil can be finally dispersed in water.
Dyes	Oil soluble organic compounds with high coloring power.	Provide distinctive or attractive color.	The organic compounds with high coloring power (dyes) dissolve to impart color.
Color Stabilizers	Certain hydroquinones, dithiocarbamates, aliphatic amines, dicyclohexylamines.	Stabilize color and prevent formation of undesirable color.	Certain chemicals can destroy color forming bodies by stopping or changing chemical reaction forming them. Sometimes accomplished by oxidation inhibitors functioning as indicated above.
Odor Control Agents	Certain oil soluble synthetic perfumes, sometimes nitro benzol.	Used to provide distinctive or pleasant odor or mask undesirable odors.	Small amounts of highly odoriferous substances impart fragrant or pleasant odor when mixed with lubricants.
Antiseptics (Bactericide or Disinfectant)	Certain alcohols, aldehydes, phenols, mecuric compounds and chlorine containing compounds.	Used to control odor, foaming, metal staining, emulsion breaking in emulsion type lubricants.	Used in soluble oils to reduce or prevent growth of bacteria causing deleterious effects in emulsion lubricants.
Pour Point	Wax alkylated naphthalene or phenol and their polymers. Methacrylate polymers.	To lower pour point of lubricating oils.	Wax crystals in oils coated to prevent growth and oil absorption at reduced temperatures.
Viscosity Index	Polymerized olefins or iso-olefins. Butylene polymers, methacrylic acid ester polymers, alkylated styrene polymers.	To lower rate of change of viscosity with temperature.	Improvers are less affected by temperature change than oil. They raise viscosity at 200°F. more in proportion than at 100°F. due to their change in solubilities.
Foam Inhibitors	Silicone polymers.	To prevent formation of stable foam.	Reduces interfacial tension so small air bubblets can combine to form larger bubbles that separate faster.

TABLE II

Effect of Time and Temperature on High Temperature Deposits Properties of Additives

High Temperature Piston Deposits Bench Test

Additive in Base Oil-B	Mgs. Deposits 525°F. 20 Hrs.	550°F. 20 Hrs.	525°F. 40 Hrs.
None	512	983	—
5 + 6 + 7	1	8	3
7 + 8	23	101	323
7 + 9	5	25	6
9	11	158	—

$$R' + O_2 \longrightarrow ROO' \text{ (peroxide)}$$
$$ROO + R'H \longrightarrow ROOH + R' \text{ (hydroperoxide)}$$

The continuous repetition of the above two reactions constitutes a so-called chain reaction whose rate is dependent on the stability of the resultant hydroperoxide. This second step of propagation becomes considerably more complex due to a so-called branching reaction sequence which results from the simultaneous reactions of the various hydroperoxides that participate or initiate some of these reactions. In the decomposition of the hydroperoxide molecule there is a break between the O—O bond to produce two free radicals as follows:

$$ROOH \longrightarrow RO' + OH' \text{ (hydroperoxide)}$$

From this branching reaction a variety of oxygen containing compounds can be formed such as water (HOH), aldehydes (RCHO), alcohols (RCH_2OH), ketones (RCOR') and acids (RCOOH). These materials may be oxidized further and react with each other to produce high molecular weight polymers. Some may be oil soluble and some may be oil insoluble and drop out as deposits. The latter may appear as resins, varnish, lacquer and coke-like deposits on metal parts of equipment. This production of long chains of repeating self propagating reactions following the initial absorption of oxygen may result in so-called auto catalytic oxidation, which may occur at an ever increasing rate. Both the initiation and propagation reactions may be activated or accelerated by presence of metal ions, heat or light.

3. The third step, termination of the oxidation reactions, may occur in one of several ways.

$$2R' \longrightarrow RR$$
$$2ROO' \longrightarrow ROOR + O_2$$

The termination can result either from the exhaustion of the oxygen supply and the exhaustion of the reactive fragments, or it may result from the formation of stable molecules as end products or the formation of free radicals that are too stable to participate in further chain reactions.

Inhibitors

Following this hydroperoxide oxidation theory, oxidation inhibitors may react in two different ways:

One type of oxidation inhibitor functions as a chain termination agent by reacting with the free radicals formed in the propagation step to form stable products which will not participate further in the reaction sequence.

The other type of oxidation inhibitor tends to passivate the metal or metal ions so that they no longer have a catalytic effect on the oxidation.

Those additives falling in the first group are considered as true antioxidants, whereas those coming under the second group are considered as catalyst poisons.

The chain terminating additives are usually organic aromatic amines, phenols or sulfides. The catalyst poisons are generally organic sulfides, phosphites or thiophosphates. These and other chemical compounds that act as catalyst poisons are often thought of as functioning by forming a protective chemical film or coating on the metal surface. This protective film may be formed on the metal surface by being physically adsorbed or chemically bonded.

Although oxidation and corrosion inhibitors are frequently referred to separately, many of the more effective inhibitors perform both functions. Those compounds containing both phosphorus and sulfur are found to be more universally effective.

The mechanism of oil oxidation and inhibitors is very complex and not completely understood because of the many factors and variations involved in it. The foregoing is one possible brief summation of it. It has been previously discussed in the January 1952 issue of *Lubrication*.

Factors Affecting Oxidation

From a more practical viewpoint, oil oxidation can be affected and is greatly dependent on the following five factors, all of which help determine the type of additive that is most suitable for a given application.

Temperature

Temperature is probably the one most important factor in oil oxidation. Any oil or other hydrocarbon will oxidize if subjected to a sufficiently high temperature for a sufficient length of time. There are obviously differences in oxidation inhibitors as to the maximum temperature at which they are stable and will inhibit against oxidation. Generally speaking, the additives in use today will withstand

TABLE III

Effect of Type of Additive on Lubrication of Different Type Metal Bearings

Additive Type Lubricant	Silver Bearings				Cu-Pb Bearings			
					MacCoull Corr., 350°F.		CRC-L-4 Eng. 280°F.	
	Silver Dish Torque Test	KOTM Test	Silver Strip Corr. Test, 325°F.	Full Scale Diesel	Mg. Wt. Loss	Rating	Gm. Wt. Loss	Rating
A	G	S	2	G	83	P	2.21	P
B	G	S	2	G	20	B	0.25	G
C	G	S	2	G	1	G	0.15	G
D*	G	S		G	35	B		P
E	B	S	2	P	85	P	3.49	P
F	P	US	2	P	1	G	0.12	G
G	P	VB	2	P	2	G	0.07	G
H	P	VB	2	P	1	G	0.11	G
I	P	US	2	P	2	G	1.21	P

*Non-Additive

Symbols: G — Good; S — Satisfactory; B — Borderline; US — Unsatisfactory; P — Poor; VB — Very Bad

Silver Strip Corr. — 1 & 2 Satisfactory, 3 and over Unsatisfactory

temperatures of up to 250°F. for a considerable length of time under many conditions. However, the length of time additive type lubricants can withstand temperatures of 300°F. and over is appreciably less. The old axiom for non-catalyzed oil oxidation that, with other conditions remaining constant, the rate of oxidation approximately doubles for every 18 to 20°F. increase in temperature really takes over. An example of this is shown in Table II.

Time

Since oil oxidation is a time temperature function, time is another factor that influences the selection of oxidation inhibitors. That is, the higher the temperature the shorter the time and, conversely, the lower the temperature the longer the time a lubricant will remain in a usable state.

Catalysts

It is generally known that some metals have a catalytic effect on oil oxidation. First of all, the rate of oxidation, especially at higher temperatures, is increased considerably by the presence of metals. Secondly, different metals and combinations of metals have different catalytic effects. Copper, iron and lead are some of the more catalytically active metals. For example, the rate of oxidation of motor oil may be increased one hundred fold by exposing it to the metals, dust and fuel combustion blow-by products found in an engine.

Thus, again, in the selection of an additive the metal or metals with which the lubricant will come in contact in service must be considered. This tends to emphasize the thought that for best performance, additives must be selected for a specific purpose. This holds for other types of additives as well as the oxidation and corrosion inhibitors.

The foregoing is illustrated by the data in Table III where it may be noted that additive oils that are good from the standpoint of Cu-Pb bearings are not necessarily good for silver lubrication and vice versa.

Contact With Air or Oxygen

Since oxidation is the chemical reaction or combining of oxygen with another element, the amount of exposure to air or oxygen is an important factor. The oxygen in oil oxidation usually comes from exposure to or mixture with air. It is only logical that the greater the amount and the more intimate the contact between air and oil, the greater will be the chance for oxidation to occur.

TABLE IV

Effect of Additives on Deposit Properties of Different Base Oils

	High Temperature Deposits Bench Test	
	Mgs. Deposits After 20 Hrs.—525°F.	
Additive No.	Base Oil X	Base Oil Y
None	970	235
7	10	10
8	30	15
10	30	15
6	115	30
1	225	445
2	355	290
9	525	310
13	750	45
4	755	595

Lubricant Characteristics

The inherent resistance to oxidation of different straight mineral oil lubricants can vary greatly due to their chemical and physical composition. The oil composition can be affected by such factors as crude source and refining processes, which can in turn affect the amounts of paraffinic, naphthenic and aromatic, as well as other components. Since lubricants varying in this respect respond differently to additives, this must be considered in the proper selection of an additive. This is illustrated in Table IV.

DETERGENT-DISPERSANTS

The main purpose of the detergent-dispersant type additives generally used in crankcase oils is to keep the engine clean. There is some difference of opinion as to the exact mechanism whereby these additives function. There is evidence to substantiate the thinking of those who feel that they function primarily as dispersants to keep oil oxidation products, fuel soot, resins and other oil insoluble materials from settling and depositing out on engine parts. However, there is also evidence which indicates that this class of additives possess true detergent characteristics, since they will remove previously formed deposits from engine metal surfaces.

There is another theory that they direct the chemical reactions occurring so as not to form oil insoluble deposits. There is also the claim that some of the more effective detergents are basic in nature so that they have a neutralizing effect on acidic oxidation products to prevent the formation of deposits.

It is quite probable that the detergent-dispersant additives act according to any one or a combination of the several ways indicated, depending on the particular conditions involved.

Some of the earlier detergent-dispersants added to oil were soluble soaps such as aluminum or calcium naphthenates or stearates. Now they are more often chemical compounds containing phosphate, phenolate, sulfonate or carboxylate groups which are usually combined with a metal such as barium, calcium potassium or magnesium. The detergents, such as metal phenates, are generally considered to function either by controlling or directing the oxidation process so as not to form oil insoluble products. The actual mechanism may be either chemical or physical in nature; the former by reaction with some of the intermediate oxidation products and the latter by adsorption or absorption of the additive on either the oil soluble particles or the metal surface to prevent deposit formation.

The function of the dispersants is believed to be primarily physical in nature. For example, dispersants such as the metal naphthenates are believed to function by peptizing sludge deposits and thus keeping them in colloidal suspension. This could apply to deposits resulting from either oil oxidation or from fuel soot or some other intermediate fuel or oil oxidation products.

There are some compounds that function primarily as detergents and others that function primarily as dispersants, but there are still others that can probably act in either way. Again, some detergent-dispersant type additive agents are most effective at high temperatures and with oxidation products formed at these higher temperatures, whereas others are more effective in minimizing deposit formation at the lower temperatures. Some of the more recently developed polymer type detergents that are claimed to be especially effective in control of low temperature type deposits, characteristic of short trip stop and go type driving, are not so effective for control of high temperature deposits.

Since engine cleanliness can be affected by deposits formed by oil oxidation, it follows that there must be a close relation between the oxidation inhibitors and detergent type additives. In view of this close relation, it is generally found more satisfactory from a practical standpoint to deal with these two types of addition agents in combination rather than individually in product development. It is desirable, of course, that the chemical compounds used for these two purposes complement rather than detract from each other. This becomes even more important because of the fact that actual effectiveness of these materials must be determined

by some performance test such as an engine test. As with other types of additives, the proper selection of a base oil is also of importance for best performance of detergent type additives. This is illustrated by the data in Table V.

In addition to their main intended function, engine cleanliness, certain detergent-dispersant type additives also often affect other properties of a lubricant. Some of these effects are beneficial, such as improved rust protection and reduced corrosion wear, whereas others are undesirable, such as increased tendency to foam and emulsify.

Effect of Detergents on Low Temperature Deposits

Low temperature type engine deposits are rather difficult to control by means of the crankcase oil. However, experience both in laboratory engine tests and service has shown that lubricants with increased detergent-dispersant characteristics are effective in reducing this type of deposit. For example, there are considerable data to show that under low temperature conditions use of oils of Series 2 detergency level results in considerably cleaner engines than with the use of an oil of MIL-L-2104 detergency level; the oils of Sup I level being intermediate.

Further reduction in low temperature type deposits can be attained by use of even more highly detergent materials such as a full strength motor detergent concentrate. The motor detergent concentrates referred to here are those prepared with additives of the same general type used in motor oils but with a considerably higher concentration, especially of the detergent components. Although such materials can be run at full strength in an engine under normal conditions, care should be exercized in their use in this manner since they have not been completely proven for all types of operating conditions.

The effectiveness of using a full strength motor detergent concentrate as the crankcase oil is illustrated in Figure 2. The engine parts shown are from a laboratory engine run at low temperature conditions which are very conducive to the formation of low temperature type deposits. The parts identified as 2A are from an engine after running the equivalent of 2,000 miles with a regular grade type motor oil. Those identified as 2B are these same parts after reassembling the engine and running the equivalent of an additional 1,000 miles using the full strength motor detergent concentrate in the crankcase. A very pronounced reduction of deposits is evident, especially the sludge deposits on parts such as oil rings, oil screen, top of cylinder head and covers where oil contact is greatest. Oil ring plugging was reduced from 50% to clean and oil screen plugging from 25% to clean. Although there was a significant reduction in the varnish type deposits as on the piston, this was less pronounced. Additional running might result in further reduction of these deposits.

This is an example of the true detergent action of the detergent-dispersant type additives, since there is a distinct removal of previously formed deposits.

The engine parts identified as 2C are from the same type of engine run as that used to produce the 2A parts, but full strength motor detergent concentrate was employed as the crankcase lubricant for the equivalent of 2,000 miles operation. In this case the engine was essentially clean except for the very small amount of piston skirt varnish.

TABLE V

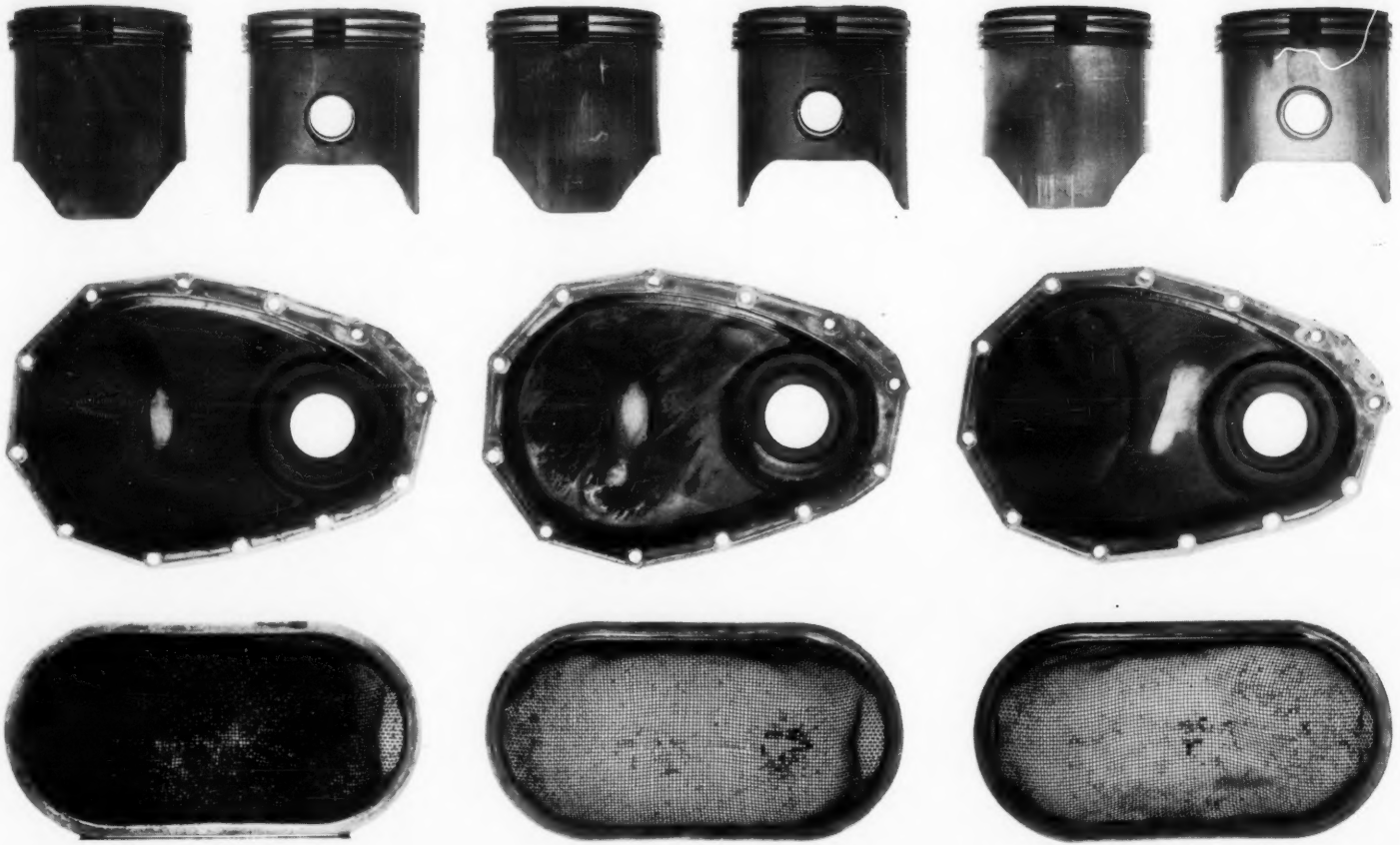
Base Oil Additive Combination

Effect of One Additive in Different Base Oils on Piston Deposits Characteristic in Supercharged Diesel

Additive No. 14 in Base Oil No.	Viscosity Index	% Ring Land Area Clean
A	26	72
B	29	70
C	72	70
D	62	64
E	87	63
F	−7	50
G	74	44
H	65	40
I	103	32
J	100	30
K	86	30
L	94	28
M	88	28
N	106	15

OILINESS, FILM STRENGTH, EP AND ANTI-WEAR AGENTS

With the ever increasing power output and capacity for a given size equipment, the unit loading and resultant pressures on critical running parts have increased. These higher loads have led to the development of so-called oiliness, film strength, extreme pressure and anti-wear agents. The basic purpose for the chemical compounds referred to by these general classes is very similar, although they may differ slightly depending on the



2A

2A — Engine Parts After 2000 Miles Low Temperature Operation Using Regular Grade Type Motor Oil in Crankcase.

2B

2B—Engine Parts From Figure 2A After 1000 Mile Operation With Motor Detergent Concentrate In Crankcase.

2C

2C — Engine Parts After 2000 Miles Low Temperature Operation Using Full Strength Motor Detergent Concentrate In Crankcase.

Figure 2 — Used Engine Parts.

specific definition used for these terms. Generally speaking, the main reason for the use of the various compounds referred to by these terms is to, (1) reduce friction, (2) prevent galling, scoring and seizure, and (3) reduce or minimize wear.

Since the complex subject of wear has been discussed rather extensively in the December 1956 issue of *Lubrication,* it will be reviewed only briefly here. Figure 3 shows the typical curve relating coefficient of friction and $\frac{ZN}{P}$ covering the three phases: hydrodynamic, quasi-hydrodynamic and boundary lubrication. Z is the lubricant viscosity, N the speed and P the load.

Referring to Figure 3, so long as lubrication remains in the hydrodynamic region there may be no real need for these special agents in a lubricant in addition to the fluid film provided by the straight petroleum oils. However, once the boundary lubrication region is reached, they are required. In the region of boundary lubrication the main friction retarding forces are due to the metal to metal contacts of the microscopically high spots of the two moving members. Under these conditions when the load is concentrated on the peaks of the surface asperities, plastic deformation of the peaks occurs until the true area of contact is sufficient to carry the load. The pressure welds formed at the tips of these peaks are sheared during the relative motion of the bearing surfaces with resultant heating and galling. Application of lubricant in a thin film to such a system changes the nature of the surface and decreases its coefficient of friction by either decreasing the number of microscopic welds or by lowering their shear strength.

Since boundary friction is a surface phenomenon, the lubricating ability of an oil blend may be changed by the use of small amounts of some types of surface active agents. These agents may function by either a chemical or physical adsorption mechanism, depending on whether or not they react with the bearing metal.

Various terms such as oiliness, lubricity, film strength, etc., have been used to describe the lubricating ability of an oil. Although a great deal of work has been done on these general subjects, the exact mechanism whereby they function is not completely understood. One reason for this is that these properties are very difficult to measure and evaluate in terms of practical application and performance because of the various factors that enter into their mechanism of functioning.

The term oiliness is generally used to indicate differences in friction resulting when different lubricants are used under the same conditions. One definition that has been proposed is that oiliness is the reciprocal of the coefficient of friction under

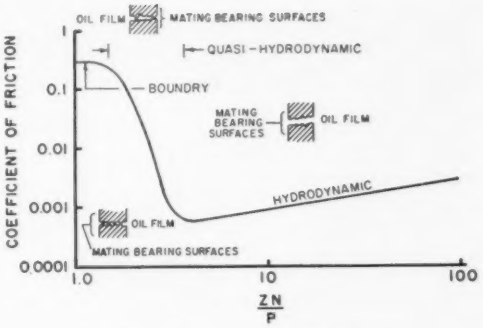


Figure 3 – Phases of Lubrication.

conditions of boundary lubrication. Certain animal and vegetable oils are superior to mineral oil in this respect, for example:

	μ	$1/\mu$
Mineral Oil	0.17	5.0
Mineral Oil + 1% oleic acid	0.10	10.1

Film strength and extreme pressure are often used as synonymous terms. The term film strength does not mean that the oil film itself prevents the metal to metal contact since the coating that keeps the metal from scoring results from the chemical reaction of the metal with the additive.

EP agents are sometimes considered a misnamed type of additive since high temperature rather than high loads present in a bearing system are actually required for their effective functioning. In some systems, localized temperatures of up to 1000°F. have been known to occur. At these high temperatures the compounds react with the bearing metals to form surface compounds having lower shear strength than the base metal, thus resulting in lower friction.

The work of some investigators indicates that anti-wear and EP agents can be distinguished from each other. The distinction is in the mechanism whereby they function. The anti-wear agents are thought to function through a chemical polishing action which can take place at relatively low temperatures, whereas the EP agents require high temperatures (probably localized) to react with the protective coating or film.

Fatty acid esters and fatty alcohols are among the commonly used oiliness agents. The EP agents used are generally compounds containing chlorine, phosphorus and sulfur such as the chlorinated waxes, organic phosphites and phosphates, and certain sulfurized organic unsaturated compounds. Certain zinc and sulfur containing compounds such as tricresyl phosphate and zinc dithiophosphate have been found to be effective anti-wear agents.

Until recent years it had been generally consid-

ered that straight petroleum oils had sufficient film strength, load carrying capacity, or lubricity for automotive engines and that they even had a sizable safety margin. However, in some of the recent engine developments, when engine output and unit loading have been greatly increased, it has been found that these straight petroleum oils are no longer adequate for all operating conditions. An example of this is shown in Figure 4. In some instances certain zinc and phosphorus containing compounds, such as zinc dithiophosphate, have been found to be very effective anti-wear agents. This is illustrated in Figure 5.

In the proper selection of any of these types of additives, the specific application for which they are to be used is very important. This is because the nature of the reaction products forming the protective coatings or films, and hence the coefficient of friction, is dependent on the specific metals involved and the surface temperatures, which in turn are dependent on other operating conditions such as load, speed and design.

RUST PREVENTIVES

The problem of rust, although chemically an oxidation process, is distinguished here from the high temperature type oxidation as encountered in engines in operation. The rusting referred to is that which occurs on ferrous parts in engines and other mechanical equipment during storage, shipment or shutdown periods. This rusting is associated with the presence of contaminants such as water or acidic materials resulting from fuel or oil oxidation. Our concern is mainly with those parts coming in contact with lubricants which cannot be protected by some coating such as paints, lacquers or varnishes.

Although straight petroleum oils have some rust protective properties, they are inadequate under the more severe conditions, and additives must be called on for assistance. Some of the chemical compounds that have been found to be effective for this purpose are the alkaline earth metal salts of high molecular weight petroleum sulfonic acids and certain complex amines and amine salts and phosphates.

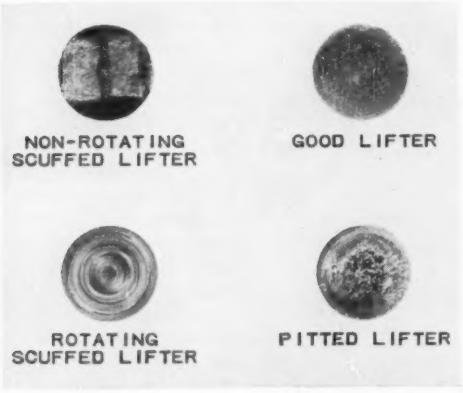


Figure 4 — Typical Valve Lifter Face Wear.

There are several ways in which rust inhibitors may function. It is thought that the amine inhibitors serve mainly to neutralize corrosive acids. However, most rust inhibitors are believed to be surface active materials which are usually long chain asymetric organic compounds with a polar group on one end. This polar group is selectively absorbed on the metal surface so that it builds up an oriented close-packed monolayer at the metal-oil interface. Since this monolayer is hydrophobic, it resists displacement by water droplets, thereby protecting the metal from attack by the water.

The effectiveness of the rust inhibitors is dependent on such factors as the strength of the polar bond between the additive and metal, the nature of the monolayer formed, the temperature conditions and base oil characteristics so that for most satisfactory results, the oil blend should be tailored to the specific conditions that exist. An example of this is the fact that some rust inhibitors require the presence of a certain amount of water before they can plate out and form a protective coating on the metal.

METAL DEACTIVATORS

Metal deactivators are sometimes considered as anti-catalysts or passivators. The functioning of the additives used for this purpose is somewhat similar to that of the oxidation-corrosion inhibitors. These additives tend to stop the catalytic effect of metal surfaces by depositing an inactive film so as to prevent contact of the lubricant with bare metal and also to prevent the metal ions from going into solution. They also tend to react with the metal ions that are in solution so as to form soluble complexes that are catalytically inactive. Both of the foregoing actions tend to reduce oxidation of the metal.

It has been found that compounds such as certain organic dihydroxyphosphines, tryalkyl and triaryl phosphites, certain active sulfur compounds, certain diamines and soaps of some metals such as tin, nickel, chromium, thallium or titanium are effective metal deactivators.

The specific compound that is most effective will depend on the metals with which the lubricant comes in contact, and also the conditions of operation such as temperature and the amount of moisture that may be present. Some compounds must be used in combination with certain oxidation in-

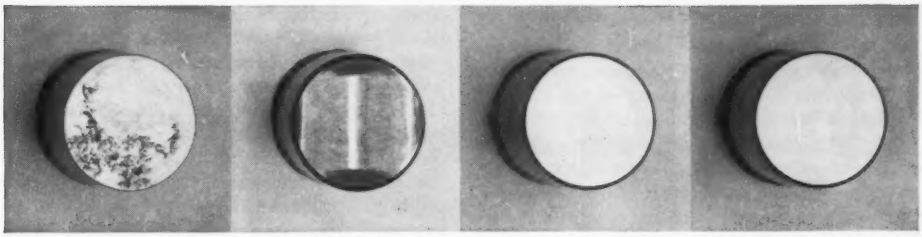


Figure 5 — Type of Reduction in Lifter Wear Effected by Anti-Wear Agents Such as Zinc Dithiophosphate.

hibitors in order to be effective as metal deactivators.

STRINGINESS OR TACKINESS AGENTS

In some lubrication applications it is necessary to increase the adhesive characteristics of the lubricating oils so that they will adhere better to the metal surfaces. This is sometimes also done to obtain protection against corrosion by forming a water repellent coating, or it may be done for certain gear and cable applications so as to maintain a lubrication film on the loaded mating metal parts. In some instances it may serve both purposes. Certain very high molecular weight polymers, as well as certain soaps, can impart this stringiness or tackiness characteristic. These materials are rather limited in their application, partly because of their limited stability. At higher temperatures (over 200°F.) and under conditions of high shear, they tend to lose their tackiness or stringiness due to breaking down into lower molecular weight polymers.

WATER REPELLENTS

Water repellency is a problem which arises primarily in connection with grease lubrication. Normally, this is controlled in the grease by the basic ingredients. However, in some instances special chemical compounds are added primarily with this function in mind. It has been found that certain surface active agents, especially those with hydrophobic (water-repellent) characteristics, are useful for this purpose. Also certain organo-silicon polymers and some aliphatic amines and hydroxy fatty acids are effective. The particular type of chemical compounds that are useful for this purpose will depend to a great extent on the type of grease involved.

It has generally been found that many of the soap thickened greases have good inherent water repellent characteristics whereas the non-soap thickened, such as those using clay or inorganic gell type thickeners are more sensitive to water. The type of additive that can be used to impart water repellent properties to the non-soap thickened grease will depend on the specific type of thickener used and also on the particular application for which the grease is intended.

EMULSIFIERS

In most lubrication applications emulsification is an undesirable characteristic. However, there are certain lubricant uses — primarily the so-called soluble oils used as coolants and lubricants for various cutting, drilling and grinding operations — where emulsification is an important characteristic. This property is usually imparted to these types of lubricants by the use of emulsifying agents. Certain soaps of fats and fatty acids, sulfonic or naphthenic acids are commonly used for this purpose. These are usually surface active chemical compounds which tend to reduce inter-facial tension so as to permit intimate mixing of oils and water to form a colloidal suspension.

DYES

There are some applications where it is found very desirable to have a distinctive color for a lubricant, mainly for identification purposes. This is of importance in those instances where leaks in the lubrication system occur on equipment where more than one type of lubricant is used. For example, on automotive engines equipped with automatic transmissions it is difficult to determine whether an oil leak is from the transmission or the engine crankcase because of the similarity in appearance of the lubricants used for these two purposes. It has been found helpful to add certain dyes which are chemical materials having high coloring power.

The main characteristic for the materials used is that they must be soluble in the lubricant to be colored. It has generally been found that the same type of dyes used in gasolines can also be used in lubricants. The quantity employed is usually considerably more for the lubricants than for gasoline because of the inherent darker colors of most lubricants. In some instances it becomes impractical to add sufficient dye to give a distinctive color, particularly in lubricants that are fairly dark in color. Although there are little data available, it would be expected that the relatively small quantities of comparatively inactive chemical compounds required would have no effect on the lubricant performance at most conditions.

COLOR STABILIZERS

Use of color stabilizers is probably of more importance with greases and fuels than some of the other petroleum products because of the undesirability of darkening and non-uniformity in color.

Many petroleum products have a tendency to turn dark in color when subjected to light or heat and oxidation takes place. It has been found that certain complex amines, such as aliphatic amines and some dicyclohexylamines, stabilize lubricants against color deterioration due to exposure to light and heat. Also, certain hydroquinones may destroy color forming bodies in some light petroleum fractions.

Although addition agents normally are not used in lubricants for this purpose, it might be expected that certain compounds could be effective in this regard. It might also be expected that anti-oxidants which minimize the formation of oxidized materials that are often darker in color would also have a beneficial effect in stabilizing color.

ODOR CONTROL AGENTS

There are some instances where lubricants are provided with a distinctive odor by the use of certain types of synthetic perfumes or highly odoriferous chemical compounds. They may be used either to provide a distinctive desirable odor for identification purposes or to mask an undesirable odor imparted by some other additives. Such materials might also be used to mask disagreeable odors that might result from normal oil oxidation or from decomposition of some of the chemical additives used. For example, some of the sulfur compounds used in EP agents may have a disagreeable odor, in either their original form or after reacting at high temperatures to perform their intended function.

ANTISEPTIC AGENTS

In the use of emulsions for various cutting and machining operations, their performance is sometimes impaired by the growth of bacteria in the emulsion circulating system. Although the soluble oils used in preparation of the emulsions are sterile in themselves, bacteria can be introduced through the water used in their preparation or through contamination while in service.

If allowed to propagate, these bacteria can cause some undesirable results such as bad odor, corrosion and emulsion breaking. To minimize the possibility of this occurring, chemical compounds that act as antiseptics or bactericides may be added to the soluble oils in their manufacture. Materials such as certain alcohols, phenols, organic mecuric and chlorine containing compounds may be effective for this purpose.

There are numerous problems associated with the selection of suitable addition agents for this purpose. Besides being effective disinfectants or bactericides, they must be compatible with the soluble oil from the standpoint of not adversely affecting its emulsifying power or its general lubricating and cooling characteristics. This requires a very delicate balance in the overall formulation of a good product. Cleanliness of the emulsion system is of extreme importance and the use of bactericides or disinfectants is not intended as a substitute for it.

It has been found that different types of bacteria can form depending on the particular conditions involved and that different chemical compounds are required for their effective control. In addition, experience has shown that some bacteria, although controllable initially by a given bactericide, tend to build up an immunity so that the particular antiseptic agent later becomes less effective or completely ineffective.

POUR POINT DEPRESSANTS

Pour point is arbitrarily defined as the lowest temperature at which an oil will flow when cooled under specific conditions. There are two known causes for observed pour point of an oil. One is that the oil cannot flow because of the crystallization of wax to form a semisolid mass. The other, pertaining more to wax free oils, is that the viscosity of the oil has become so great that it ceases to flow. Both of these effects can be minimized or countered by refining processes and the use of certain base stocks. However, this can be costly from the standpoint of the added processing, reduced yields and limiting use of available stocks which may have other more desirable characteristics.

An equivalent effect can be accomplished more economically by the use of addition agents generally referred to as pour depressants. Some high molecular weight polymers with about the same solubility in the oil as the wax have been found to be very effective for this purpose, especially for the wax containing oils. Among the materials used for this purpose are wax alkylated naphthalene and wax alkylated phenol and their polymers and polymers of methacrylic acid esters.

These additives are believed to function by forming a protective or insulating film on the surface of the wax crystals as they are formed thus preventing the separate wax crystals from adhering to each other to form a matrix or sponge structure which could trap the oil and hold it in a rigid semisolid condition.

The problem of pour point reversion is sometimes encountered with lubricants containing pour depressants. The pour point reversion occurs when

the temperature cycle is such that wax crystals are allowed to exist for an extended period of time. This usually occurs when the temperature rises above the pour point but not sufficiently for all the wax crystals to completely go into solution. As a result, the protective film formed on the wax crystals by the pour depressant becomes ineffective, and the interlocking of wax crystals is gradually increased until the pour point of the base oil is reached. Certain pour point depressants are more prone to this than others.

Since both some of the VI improvers and pour depressants are some type of high molecular weight polymers, certain of these compounds can serve both purposes. The reverse of this is not necessarily true since, all VI improvers will not act as pour depressants or, similarily, all pour depressants will not act as VI improvers. As in the case of VI improvers, certain pour depressants are more effective in some base oils than others. Similiarly, certain base oil pour depressant combinations may be more suitable for some types of service than others.

VI IMPROVERS

In view of the increased temperature range over which mechanical equipment is required to operate, VI improvers were developed for use in various lubricants. The more commonly used VI improvers are generally long chain high molecular weight polymers of compounds such as alkyl styrenes, butylenes or esters of methacrylic acids. The compounds found to be effective as VI improvers are generally considerably more viscous than the lubricating oils in which they are used. They may have extrapolated viscosities ranging from 25,000 to 60,000 SUS at 100°F. The materials when added to oil have a considerable effect on the temperature-viscosity relation commonly referred to as viscosity index (VI). This is an empirical scale developed by the petroleum industry to give a simple numerical expression to the relation of an oil's viscosity to its temperature.

There is some controversy as to just how VI improvers function. One of the popular explanations as to how this type of additive functions is that the long chain polymers are only slightly soluble in oil at low temperatures so that most of the material is in colloidal suspension and has little effect on its viscosity. As the temperature increases, more and more of these polymers go into true solution so that their relatively high viscosity contributes more and more to the viscosity of the oil blend. Thus, at low temperatures the viscosity is essentially that of the base oil, while at higher temperatures the viscosity varies with the amount of polymer that goes into true solution. This in turn can vary, within certain limits, with the concentration and solubility of the VI improvers used.

TABLE VI

Shear Stability of VI Improvers

Laboratory Test Where Oil is Subjected to High Rate of Shear by Forcing Through Small Orifice

Base Oil plus VI Imp. %	Type	New Oil Visc. at 100°F.	% Decrease After Shearing Visc. 100°F.	210°F.	V.I.
None		202	0	0	0
10	A	462	31	25	42
10	B	461	36	30	7
10	C	476	46	40	10
10	D	407	11	10	1
2	C	243	13	9	16
6	C	346	31	25	17

Since the effectiveness of VI improvers is related to their solubility in the base oil, chemical composition and other characteristics will, of course, also be determining factors in their effectiveness. It follows that some VI improvers are more effective in certain base oils than others, and this must be considered carefully in the selection of a VI improver. This also helps determine the maximum or optimum concentration of a VI improver that can be used in a given base oil.

One problem that has been encountered in service is that oils containing appreciable quantities of these high molecular weight polymers tend to undergo a viscosity change when subjected to very high rates of shear. This change can manifest itself as a change in VI or a change in viscosity, or both. A change in VI means a greater change in viscosity as measured at one temperature than at another. However, there also may be a proportional change in viscosity at all temperatures so that the VI is unaffected. Insufficient data are available at this time to show definitely if the viscosity and/or VI change is due to the actual shearing down or breakup of the polymer molecule into smaller lower molecular weight shorter chain polymers or to the effect that the vigorous agitation has on the dispersion or colloidal suspension of the polymers in the oil blend.

There are great differences among VI improvers insofar as their shear stability and effectiveness is concerned. Generally speaking, the greater the concentration of any given VI improver used, the greater will be the amount of shear down that can occur in the oil blend. This is illustrated by the data in Table VI. The foregoing all leads to the fact that the specific service for which a product is

intended must be considered carefully in order to make the best selection of VI improvers and base oil combination.

ANTI-FOAM

Foaming is a problem sometimes encountered with lubricating oils. Any oil will tend to foam if agitated with air vigorously enough. The amount of foaming can vary considerably for different types of oils and, of course, with the conditions under which it is used. As a general rule, the more viscous oils will have a greater tendency to foam more since they are better able to hold air bubbles and thus form a more persistent foam. Also oils have a tendency to foam more at the higher temperatures, but the foam will be less persistent because of the lower viscosity of a given oil as temperature increases. Additives necessary to attain some of the desired characteristics in oils may increase their foaming tendency because of the mechanism whereby they function.

Although there are a number of chemical compounds that have anti-foaming properties, probably the most widely used are the silicone polymers. Rather low concentrations of these materials are very effective in accelerating the rate at which foam breaks. There is some indication that these compounds act primarily to increase the rate at which foam breaks and that they do not necessarily reduce the air entrained in the oil while circulating or being agitated.

One popular theory on how the anti-foam agents function is that they are actually insoluble in the bulk oil and are held in colloidal suspension. In this way they are able to reduce the interfacial tension around the small air bubblets so that when they come near each other, they tend to combine to form larger bubbles which will rise to the surface more rapidly and break. This is shown schematically in Figure 6. Thus, the rate of air release will be nearer the rate of entrainment so that there is less air to form foam.

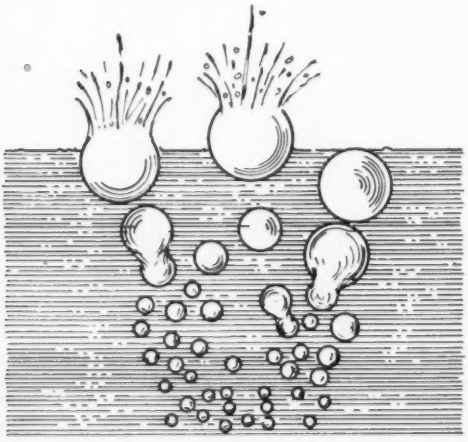


Figure 6 — Anti-Foam Action to Combine Small Bubblets to Form Larger Bubbles That Separate Faster.

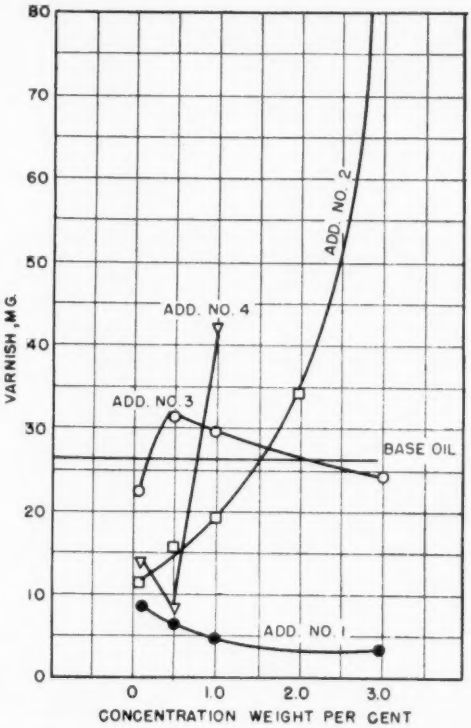


Figure 7 — Effect of Additive Concentration on Deposits High Temperature Oxidation Test 338°F., 40Hr.

The amount of anti-foam agent required will depend on some of the other properties of the lubricant and to a large extent on the operating conditions at which the lubricant is used. For example, conditions in an automatic transmission are considerably more conducive to foaming than those in an engine crankcase. Accordingly, more inhibition against foaming is required.

BALANCED ADDITIVE COMBINATIONS

Perhaps the one factor that contributes most to making the development of good finished additive type lubricants very complex is the necessity for a well-balanced additive combination. Few finished lubricants are in extensive use today which contain an additive agent that affects only one of its properties. It is more common for lubricants to contain additives that will affect at least two and sometimes

as many as 10 or 12 of its characteristics.

Automatic Transmission Fluid Type A is an example of one of the more complex lubricants from an additive standpoint. To meet the requirements specified for this fluid it is generally necessary that it contain chemical addition agents of the following types: oxidation-corrosion inhibitor, detergent-dispersant, VI improver, pour point depressant, anti-foam agent, EP, anti-wear, oiliness and anti-friction agents and rust preventives. In addition, it must be non-toxic, not possess any undesirable odor or form such on use and not adversely affect various materials such as both ferrous and non-ferrous metals, various types of synthetic rubber and other types of seals, cork, krafelt or other clutch and band facing materials. These must not be affected adversely over a wide temperature range. Finally, the fluid must work satisfactorily in various types of automatic transmissions in service as well as in accelerated laboratory and proving ground tests.

Since the possibility exists that any chemical addition agent, whose primary purpose is to affect a given property, can also react or affect any other chemical addition agent, the number of possible problems that might arise increases greatly for every chemical compound incorporated in a given lubricant. Considerable testing is required to determine the optimum concentration of a single additive material both from the standpoint of best performance and also as to its economic aspects. This again becomes more complex, and the amount of work required is increased considerably when combinations of many chemical addition agents are involved. The data in Figure 7 show the effect of concentration of various additives on oil deposit forming characteristics.

Experience has shown that some chemical addition agents can have either a synergistic or anti-synergistic effect on each other; the former being desirable and the latter undesirable. Synergism is when the effect of the combination of two components, as measured in a given test, is greater than the sum of the effects resulting from use of each of the components individually. Conversely, anti-synergism is when the combined effect is less than the sum of the individual component's effects. These effects are quite common in oxidation-corrosion inhibitors and detergent combinations and add greatly to the complexity of developing additive type lubricants containing more than one chemical addition agent. Examples of synergistic and anti-synergistic effects are shown in Table VII.

TABLE VII

Synergistic and Anti-Synergistic Effects

Additive in Base Oil-A	High Temperature Piston Deposits Bench Test 525°F. for 20 Hours — Mgs. Deposit: Synergistics	Anti-Synergistics
None	200	
1	186	186
2	162	162
3		172
4	376	
1 + 2 + 3		660
1 + 2 + 4	4	
	MacCoull Corrosion Test 350°F.—10 Hrs. Cu-Pb Brg. Wt. Loss, Mg.	
15		7
16		4
17		8
15 + 16		23
16 + 17		28
	Hours to Reach Neut. No. of 2.0 in Catalyzed Oxidation Test	
18	350	
19	250	
18 + 19	1800	

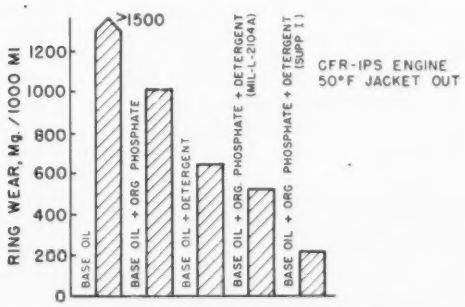


Figure 8 — Ring Wear Reduction by Additives.

The mechanism involved and the reasons for these synergistic effects are not completely understood. However, the occurrence of these effects and their magnitude is dependent to some extent on the environment and conditions under which the additives are used.

The effectiveness and importance of properly balanced additive combinations for a specific application or problem is illustrated by the data in Figure 8. Although detergent type additives are generally not considered as anti-wear agents, their effect on some types of low temperature wear is very evident from these data. However, this does not necessarily

mean that they will be effective as anti-wear agents under other conditions.

PERFORMANCE TESTING

Performance testing of additives in lubricants is a subject in itself and beyond the scope of the present article. However, it must be mentioned briefly, at least, since the ultimate development of any satisfactory additive type lubricant is dependent upon a rigorous and complete testing program. Although the nature and extent of the tests conducted will be specific for each type of lubricant and the properties involved, the same basic pattern is generally followed.

In the first stages, countless laboratory bench tests are used to screen the additives under consideration. These tests serve a very useful purpose since they usually have the advantage of affording very close control of all the operating conditions. They are especially valuable in research and development of additive type lubricants, since rather small differences attributable to additive effects on certain characteristics can be determined. The data obtained from them are generally on a relative basis, however, so that it is necessary to develop adequate correlation data with service in order to make them more useful.

The formulations which appear promising are then subjected to simulated service tests which have been designed to reproduce a certain condition or set of conditions to which the lubricant will be exposed in actual service. In tests of this nature, the conditions are generally very carefully controlled. In spite of this, there are certain variations that are inherent in the equipment and which can cause deviations in performance. These must be taken into consideration in the interpretation of the test results. At present there is no known substitute for experience in interpreting such test results.

In the development of additive type lubricants for use in automotive equipment, road tests have been found very useful. These may be considered as semi-controlled service tests since they are usually run using various makes of equipment and under different types of conditions. These tests are controlled from the standpoint that all vehicles used for direct comparison are checked to see that they are in the same mechanical condition at the start of the test and all vehicles are run under the same test conditions. In such tests a reference lubricant of known service performance is usually included for further direct comparison and interpretation of test results.

One approach that has been found useful in this type of testing is to run one group of vehicles at high temperature, high speed conditions and another at low temperature, low speed stop and go type of conditions. Covering the two extremes of test conditions normally encountered in service generally provides adequate assurance that the products will also perform satisfactorily at intermediate conditions.

For lubricants intended for other types of equipment and uses, the test lubricant being developed may be used in a few units in actual service. For example, a few railroad diesel engines or a fleet of buses or trucks or tractors or a few paper mill machines may be operated on the test lubricant for direct comparison with the performance of similar units operating on the products which have been in use. In these field tests close checks are maintained on the test units to obtain information on the improvement in performance resulting from the product under development as well as to detect abnormal or adverse conditions that might arise. Such tests are run under normal service conditions without special control except for the observations just indicated.

As a last step before a newly developed product is placed into full scale marketing, it may be further checked in actual service conditions by first distributing it in a limited area for a short period. The performance of the product in the limited area is checked very closely and carefully. This is done primarily to give assurance that no adverse conditions will develop due to occurrence of a peculiar combination of uncontrolled conditions which could not be predicted by the various laboratory and field tests previously conducted.

SUMMARY

The changing lubrication requirements that have accompanied many developments in engines and other mechanical equipment over the years have been met by corresponding developments in lubricants. Many of the improvements to meet these requirements have been made possible through the extensive use of various chemical addition agents. It is expected that developments of this type will continue in the foreseeable future in the field of engine lubricants and also expand more in other industrial type lubricants. As has been emphasized in the various preceding sections, a tremendous amount of work is required on the part of both the additive manufacturers and the lubricant producers to assure the various users of lubricants satisfactory products for their specific uses. The extensive experience and reputation of the suppliers of these products is the consumers best assurance of quality products that will provide satisfactory performance. The continued close cooperation between the equipment manufacturers and lubricant suppliers is further assurance of continued satisfactory lubricant performance in newly developed equipment as it is placed into service.

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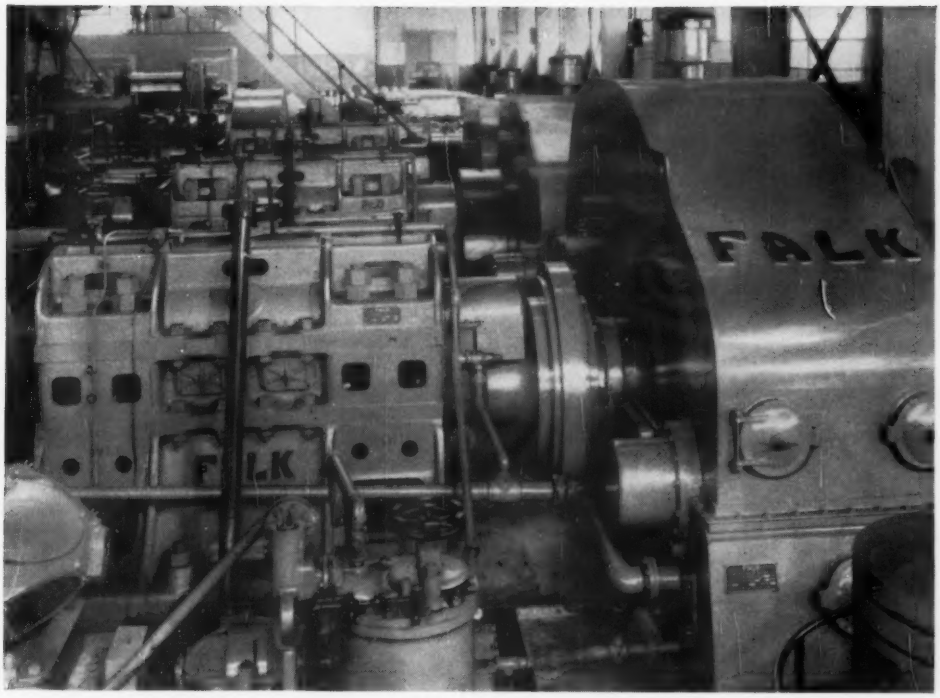

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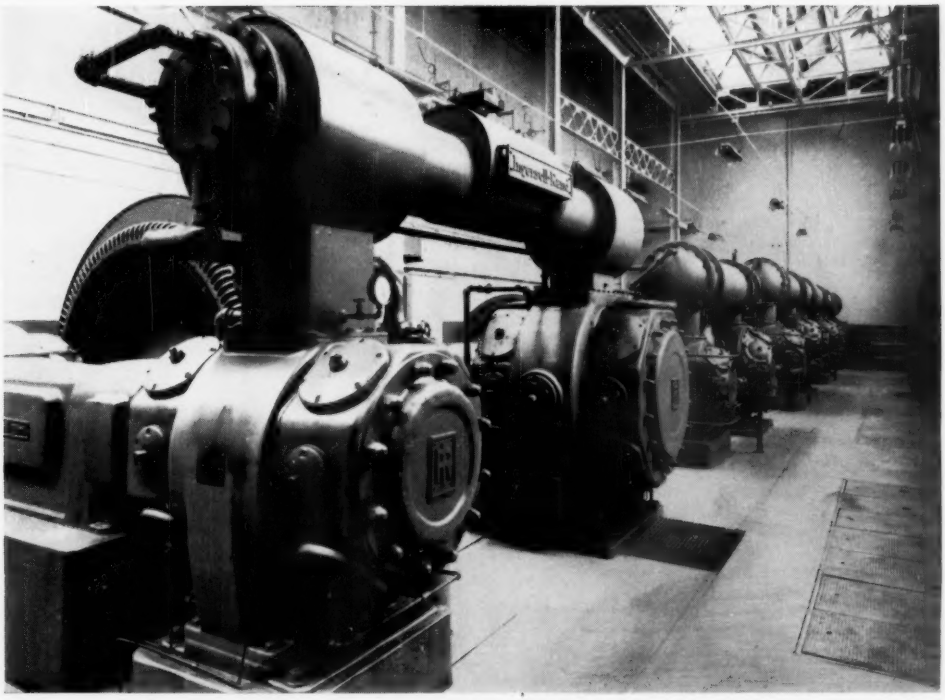

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